

748

REPORT ON MICROANALYTICAL DETERMINATION OF SULFUR

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Last year's collaborative studies (1) on the determination of sulfur indicated a preference for the Carius and catalytic combustion procedures over the Parr bomb method, both from the data obtained last year and also from the relative number of analysts using the Parr bomb procedure. Consequently, the objective of this year's work was to test the two preferred procedures, the Carius and the catalytic combustion, to see if they were sufficiently accurate and precise to warrant recommending them for adoption, first action. The details of the procedures were based upon the results of the statistical study of last year's data. In those instances where the results showed no preference for one technique or condition over another, the condition or technique included in this year's method was either the one most frequently used by the collaborators, the more simple technique, or the more easily attained condition. The same two samples, namely, benzyl-isothioureia hydrochloride and sulfanilamide, were sent to each collaborator with copies of the tentative procedures, which they were requested to follow as closely as possible. With but few exceptions, the collaborators adhered closely to the specified procedures. Three analysts used the one-piece Pregl combustion tube instead of the two-piece Beazley type and two used a Grote absorber instead of the spiral, but in no case was the change believed to be important, because neither of these variations affected the results significantly in last year's

study. Collaborator 2 used a more rapid oxygen flow rate in the catalytic combustion than was specified but used the Grote absorber to counteract this. He also used a photometer rather than a visual comparator to determine the end point in the titration of the sulfate. Collaborator 30 used an electroprecipitator instead of bromine for absorbing the sulfur trioxide. These variations had little or no effect on the results; therefore their data was used in the statistical calculations.

The procedures for the Carius and the catalytic combustion methods for the micro determination of sulfur which were sent to the collaborators for study this year were as follows:

CARIUS COMBUSTION METHOD

REAGENTS

- (a) *Fuming nitric acid*.—Reagent grade, sp. gr. 1.50.
 - (b) *Sodium chloride*.—Reagent grade, fine crystals.
- For volumetric determination:
- (c) *Barium chloride solution*.—Ca 0.02 *N*, standardized by titrating 5–7 mg freshly dried K_2SO_4 , A.C.S. specification (weighed to the nearest 0.01 mg) by same procedure used for the sample titration. Correct titration for indicator error by blank run.
 - (d) *Potassium sulfate*.—A.C.S., powd. and dried.
 - (e) *Sodium hydroxide*.—Ca 0.1 *N*.
 - (f) *Hydrochloric acid*.—Ca 0.02 *N*.
 - (g) *Phenolphthalein*.—0.5% soln in 50% alcohol.
 - (h) *Sulfate indicator*.—"T.H.Q." prepd indicator (Betz Laboratories, Philadelphia, Pa.).
- For gravimetric determination:
- (i) *Dilute hydrochloric acid*.—Add 1 ml concd HCl to 300 ml distd H_2O .
 - (j) *Barium chloride solution*.—10%.
 - (k) *Barium chloride solution*.—Ca 0.1 *N* (for alternate grav. detn.).

APPARATUS

- (a) *Combustion tubes*.—Use clean, 240 ± 10 mm by 13 ± 0.7 mm O.D. standard wall pyrex glass tubes or 210 ± 10 mm by 13 ± 0.8 mm O.D. pyrex glass tubes with 2.3 ± 0.3 mm walls (see Table 1) free from flaws and with rounded seal at the bottom.
- (b) *Furnace*.—Elec. with capacity of 4 or more tubes held at angle of ca 45° . Furnaces must maintain temp. of 250 ± 10 or $300 \pm 10^\circ C$. for 5 or more hr, with no more than 5° difference between any two points on a tube, or 5° difference between similar points on any two tubes. Furnace must have variable resistor or other device to adjust furnace to desired temp. Open end of furnace wells must have safety device to retain glass in furnace should tube explode, and device must be provided for removing individual tubes from wells.
- (c) *Crucible and filter stick* (for gravimetric determination).—Porcelain crucible, ca 15 ml capacity, with black inside glaze weighing about 10 g. Porcelain filter stick with unglazed bottom, weighing about 2 g (3).
- (d) *Filter tubes* (for alternate gravimetric determination).—Micro filter tube with medium porosity fritted disc and capacity of 3 ml.
- (e) *Titration assembly*.—5 ml buret graduated in 0.01 ml; rectangular titration

cell ca 2×4×5 cm with min. capacity of 50 ml; and standard orange-red glass color filter having 37% transmittancy at 550 m μ . (Arthur H. Thomas Co., Philadelphia, Pa., Cat. No. 9324-H.) Cell and filter are placed side by side on milk glass window illuminated from below, preferably by fluorescent light. The light source must be masked so that only the cells and filter are illuminated.

SAMPLE

Using microchemical balance, weigh 5–20 mg sample contg not less than 0.75 mg S or using semimicrochemical balance, weigh 10–20 mg sample contg not less than 0.75 mg S for volumetric analysis, or 1.5 mg S if gravimetric procedure is to be used.

Solid samples.—Weigh by difference in charging tube.

Viscous liquids and gummy solids.—Weigh in micro porcelain boat.

Volatile liquids.—Weigh in ca 5 cm long, 1–2 mm I.D. sealed glass tube with capillary tip. Break off tip of capillary before placing in combustion tube sealed end down.

DETERMINATION

Place weighed sample in combustion tube, add NaCl 100% in excess of amount equivalent to S in sample, and add 0.3 ± 0.03 or 0.5 ± 0.05 ml fuming HNO₃ depending on type of combustion tube (see Table 1). Using blast lamp and holding tube at 30–40° angle, seal tube at a distance from bottom so that sealed tube will have

TABLE 1.—Combustion tubes

COMBUSTION TUBE	WALL THICKNESS	O.D.	LENGTH	LENGTH OF SEALED TUBE BETWEEN BOTTOM AND START OF TAPER AT SHOULDER	VOL. HNO ₃ (SP. GR. AT 60°F., APPROXI- MATELY 1.5)	TEMP. °C.
	mm	mm	mm	mm	ml	
Heavy-walled	2.3 ± 0.3	13 ± 0.8	210 ± 10	150 to 175	0.5	250
Thin-walled	1.2 ± 0.2	13 ± 0.7	240 ± 10	180 to 210	0.3	300

length shown in Table 1. Rotate tube slowly in flame until wall thickens, pull out and seal off narrow neck of tube. Wall of seal should not be less than $\frac{3}{4}$ the thickness of tube wall. (If sample and nitric acid react at room temp., immediately cool bottom of tube in ice-H₂O or dry ice-acetone bath.) Immediately place tube in furnace. Heat tube for 5 hr at 250 or $300 \pm 10^\circ\text{C}$. (see Table 1).

Observe the following precautions before and during opening of combustion tubes. (a) Place asbestos glove on hand used to hold small burner or hand torch; (b) protect the face by transparent face mask or work behind safety shield; (c) be certain tube has cooled to room temp.; (d) force tip of tube about 2 inches out of furnace well; (e) gently flame end to drive all acid from tip and upper walls; and (f) soften tip with small hot flame until pressure in tube is released by blowing out softened glass.

Remove vented tube from the furnace and cut off constricted end by scratching tube with file 0.5–1" below shoulder of open end, moisten scratch, and touch with tip of very hot glass rod. Remove end of tube with care and fire polish if gravimetric procedure is to be used to avoid contaminating ppt. with glass splinters. Transfer

contents of tube to 50 ml beaker, rinsing tube 4–6 times with 3–5 ml H₂O. Evap. to dryness on steam bath.

*Volumetric determination.**—Dissolve residue in 10 ml distd H₂O. Pour soln into titration cell, add one drop phenolphthalein indicator, make just alk. with 0.1 N NaOH, then acid with 0.02 N HCl adding one drop in excess. Add ca 0.15 g of "T.H.Q." indicator, stir to dissolve, rinse beaker 2 or 3 times using sufficient ethanol so that final soln is ca 50% ethanol. Titrate with standard BaCl₂ soln from 5 ml burette graduated in 0.01 ml until stable color of the soln immediately after stirring matches standard glass color filter. (Make certain end point taken is real and not pseudo end point which will fade on standing 1–2 min.) Run blank on reagents and correct titration value.

CALCULATION

$$\frac{(\text{ml BaCl}_2 - \text{blank}) \times N \times 16.033 \times 100}{\text{Sample wt (mg)}} = \% S$$

Gravimetric determination.—Dissolve residue in 3–5 ml of 1:300 HCl, pour into previously ignited and weighed porcelain crucible. Rinse beaker with four 1–2 ml portions of 1:300 HCl and place crucible on steam bath until soln is near boiling point. (If total vol. exceeds 10–11 ml evap. to this vol.) Add 1 ml 10% BaCl₂, digest for at least 0.5 hr and cool to room temp. Connect porcelain filter stick previously ignited and weighed with crucible to arm of siphon with rubber tubing, the other arm of siphon being connected to suction flask through a rubber stopper. Lower filter stick into crucible, draw off soln, rinse ppt., walls of crucible, and filter stick alternately with 1–2 ml portions of 1:300 HCl and ethanol, drawing off as much liquid as possible. Carefully detach filter stick, place in crucible and wipe outside of crucible with moist chamois or cheesecloth. Dry in oven at ca 110°C. for 10 min., then ignite in muffle furnace at 700°C. for 10 min. (Ignition may be carried out by setting crucible containing filter stick in larger porcelain crucible and heating larger crucible to dull red heat with Meker burner.) Cool on metal block for 20 min. (or in desiccator for 1 hr) and weigh. Make blank run on reagents.

CALCULATION

$$\frac{(\text{wt BaSO}_4 - \text{blank}) \times \frac{S}{\text{BaSO}_4} \times 100}{\text{Sample wt}} = \% S$$

Alternate gravimetric determination.—Dissolve residue in 10 ml distd H₂O. Add 1 drop concd HCl, heat to near 90°C. on steam bath, add 5 ml 0.1 N BaCl₂, digest for at least $\frac{1}{2}$ hr and cool to room temp. Place previously washed, dried, and weighed filter tube (medium porosity) in one-hole stopper in suction flask, connect small funnel (1–1.5" diam.) to filter through small rubber stopper with funnel tip protruding ca 0.25". Transfer ppt through funnel to filter tube, rinsing beaker and funnel alternately with 1:300 HCl and 95% ethanol using 2 or 3 ml portions. Remove funnel, rinse tip and stopper with alcohol, then rinse filter and ppt with 1:300 HCl, then with alcohol. Wipe outside of filter tube with moist chamois (or cheesecloth) and dry either at 135°C. in air oven or at 80° in vacuum oven for 0.5 hr, place in open air by balance until cooled to room temperature (20 min.) and weigh. Handle dry tube with clean chamois finger cots or tweezers. Make blank run and subtract any correction from weight of sample ppt.

* Volumetric detn. cannot be used if sample contains phosphorus.

CALCULATION

$$\frac{(\text{wt BaSO}_4 - \text{blank}) \times \frac{S}{\text{BaSO}_4} \times 100}{\text{Sample wt}} = \% S$$

CATALYTIC COMBUSTION METHOD

REAGENTS

Use reagents of the Carius combustion method above, and

(a) *Bromine water*.—Satd aq. soln of Br stored in glass-stoppered bottle.

For gravimetric determination:

(b) *Hydrogen peroxide solution*.—Dil. 20 ml reagent grade 30% H_2O_2 with 80 ml distd H_2O .

APPARATUS

(a) *Oxygen supply*.—Use O_2 pressure cylinder with 2 stage reducing valve having needle valve control on low pressure side, or any other source which will supply pure O at 12–15 ml/min.

(b) *Purification train*.—If O is not free from S-contg gases, purify by passing gas through a tube containing first Dehydrite, then Ascarite.

(c) *Combustion tube*.—Quartz (or Vycor) with dimensions shown in Figure 1.

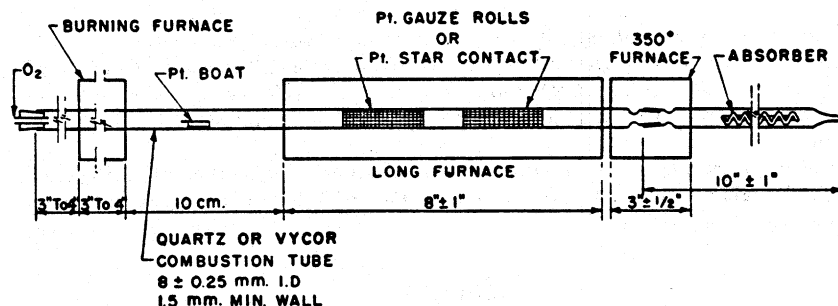


FIG. 1.—Combustion Assembly for Sulfur Determination by Catalytic Method.

(d) *Absorber*.—Beasley type spiral connected to combustion tube by ground joint.

(e) *Catalyst*.—2 Pt star contacts (3) or 2 Pt gauze rolls made from 5 cm squares of ca 50 mesh gauze. Rolls to have diam. within 1 mm of I.D. of combustion tube.

(f) *Furnaces*.—Elec. or gas, elec. preferred, both providing temp. inside combustion tube of at least 750°C. and preferably 800°C. or over. Sample burner operated mechanically or manually, the former being preferred. Short furnace for ground joint, preferably elec., to operate at ca 350°C. Rate of motion of sample burner 0.5 cm/min.

(g) *Titration assembly*.—See Carius combustion method above.

(h) *Crucible and filter stick (for gravimetric determination)*.—Porcelain crucible, ca 15 ml capacity, with black inside glaze, weighing about 10 g. Porcelain filter stick, with unglazed bottom, weighing about 2 g (3).

(i) *Filter tubes (for gravimetric determination)*.—Micro filter tube with medium porosity fritted disc and capacity of 3 ml.

SAMPLE

Use sample described under Carius combustion method above.

DETERMINATION

Clean catalysts by boiling 10 min. in ca 6 *N* HNO₃ and flaming over non-luminous flame, using Pt tipped tweezers. Place catalysts in combustion tube and set tube in furnaces as shown in Figure, heat long furnace to at least 750° and preferably 800°C. or over.

Moisten entire spiral of absorber by drawing into it with gentle suction 5–10 ml of Br water for volumetric analysis or 5–10 ml. of H₂O₂ for gravimetric analysis. Care must be exercised to keep ground joint dry. Drain excess soln from absorber and attach to combustion tube with ground joint in 350° furnace. Place sample in combustion tube 5 cm from long furnace, connect O source and adjust flow to 12–15 ml/min. using flow meter or calibrated bubble counter.

Heat sample burner to at least 750°, preferably 800°C. or over, bring to 5 cm from sample and move over sample area at rate of 0.5 cm/min. (burning time 20 min.). Continue sweeping with O for 10 min. (total combustion time 30 min.). Disconnect absorber, allow joint to cool 3–5 min.

*Volumetric determination.**—Rinse contents quantitatively into 50 ml Erlenmeyer flask using 15–20 ml H₂O. Rinse outside of absorber tip. Add 5 drops Br water, boil until Br is dispelled and cool under tap. Continue as under Carius combustion method beginning with "Pour solution into titration cell, . . ."

Gravimetric determination.—Rinse contents of absorber quantitatively into previously ignited and weighed porcelain crucible using five 2 ml portions of 1:300 HCl. Place crucible on steam bath and heat to near b.p. (If total vol. exceeds 10–11 ml evap. to this vol.) Continue as under Carius Method beginning with "Add 1 ml 10% BaCl₂. . ."

Alternate gravimetric determination.—Rinse contents of absorber quantitatively into 50 ml beaker with 1:300 HCl using 4–6 rinses of 3–5 ml each. Rinse outside of absorber tip. Continue as under Carius Method beginning with "heat to near 90°C. . ."

RESULTS

The tabulated results reported for both samples by the various procedures are shown in Table 2 in which *n* is the number of values reported by the collaborator; \bar{x} , the mean of his values; *s*, the standard deviation of his values; *n_x*, the number of \bar{x} values used; $\bar{\bar{x}}$, the mean of the \bar{x} 's; *s_x* the standard deviation of the means; and \bar{s}_x , the mean of the standard deviations.

Twenty-one collaborators reported 22 sets of data for sample 1 (benzylisothiurea hydrochloride) and twenty-three sets of data for sample 2 (sulfanilamide). Values reported by collaborator 66 could not be used in the statistical calculations because they were obtained by a method similar to that described by Stragard and Safford rather than by the procedure being tested. They were included in the table for comparison, however. The 21 and 22 sets of data used in the calculations represented 97 and 109 determinations, respectively, for samples 1 and 2. The grand

* Volumetric detn. cannot be used if sample contains phosphorus.

TABLE 2.—Data and conditions for sulfur determinations

COLLAB. NO.	n	\bar{x}	s	\bar{x} -THEORY	CARIUS	CATA- LYTIC	TITRI- METRIC	GRAVI- METRIC	ALT. GRAV.
Sample 1. Benzyl-iso-thiourea hydrochloride (15.82% S)									
0	7	15.80	0.08	-.02	x		x		
1	4	15.80	0.10	-.02		x	x		
2	3	15.65	0.03	-.17		x	x		
9	6	15.87	0.10	+.05		x	x		
15	4	15.73	0.05	-.09	x		x		
17	8	15.82	0.06	.00	x		x		
29	3	15.41	0.19	-.41	x			x	
30	8	15.86	0.12	+.04		x	x		
37	4	16.14	0.28	+.32	x			x	
40	4	15.77	0.12	-.05		x		x	
45	6	15.54	0.23	-.28		x	x		
46	4	15.92	0.13	+.10	x		x		
49	8	15.71	0.09	-.11	x		x		
50	4	15.83	0.04	+.01	x		x		
51	2	15.60	0.17	-.22		x	x		
59	4	15.73	0.22	-.09	x				
60	4	15.84	0.05	+.02		x	x		
60'	4	15.92	0.10	+.10	x				x
65	4	16.09	0.05	+.27		x			x
66*	(4)	(15.89)	(0.20)	(+.07)		(x)		(x)	
69	4	15.89	0.15	+.07		x		x	
8	2	15.93	0.09	+.11	x			x	
$n_{\bar{x}}$	97	21	21		11	10	13	5	2
\bar{x}		15.81		-0.01	15.81	15.79	15.77	15.82	16.01
$s_{\bar{x}}$		0.17			0.17	0.16	0.11	0.11	0.12
s_s			0.12		0.12	0.11	0.10	0.17	0.08
Sample 2. Sulfanilamide (18.62% S)									
0	7	18.54	0.16	-.08	x		x		
1	4	18.61	0.07	-.01		x	x		
2	3	18.66	0.04	+.04		x	x		
9	6	18.70	0.10	+.08		x	x		
15	4	18.62	0.04	.00	x		x		
15'	6	18.64	0.09	+.02		x			x
17	8	18.61	0.04	-.01	x		x		
29	3	18.31	0.06	-.31	x			x	
30	7	18.63	0.11	+.01		x	x		
37	4	18.78	0.17	+.16	x			x	
40	6	18.65	0.11	+.03		x		x	
45	6	18.48	0.06	-.14		x	x		
46	5	18.77	0.09	+.15	x		x		
49	8	18.50	0.12	-.12	x		x		
50	8	18.65	0.07	+.03	x		x		
51	2	18.72	0.04	+.10		x	x		
59	4	18.60	0.15	-.02	x				
60	4	18.66	0.09	+.04		x	x		
60'	4	18.52	0.19	-.10	x				x
65	4	18.71	0.18	+.09		x			x
66*	(4)	(18.33)	(0.30)	(-.29)		(x)		(x)	
69	4	18.80	0.19	+.18		x		x	
8	2	18.59	0.01	-.03	x			x	
$n_{\bar{x}}$	109	22	22		11	11	13	5	3
\bar{x}		18.62		.00	18.59	18.66	18.63	18.63	18.62
$s_{\bar{x}}$		0.11			0.13	0.08	0.08	0.20	0.10
s_s			0.10		0.10	0.11	0.08	0.11	0.15

* Data obtained by Stragand-Safford method and not used in statistical calculations.

mean, \bar{x} , for sample 1 was 15.81 per cent sulfur as compared with a theoretical value of 15.82 per cent. The standard deviation of the means, $s_{\bar{x}}$, was 0.17 per cent, whereas the average of the s values was 0.12 per cent, indicating that the precision within laboratories was better than that between laboratories for sample 1. The \bar{x} for sample 2 was identical with the theoretical value, 18.62 per cent sulfur, and the $s_{\bar{x}}$ value was 0.11 per cent which agrees closely with the average s value, 0.10 per cent, indicating approximately the same precision within and between laboratories.

Since there was a choice as to methods (Carius or catalytic combustion) and as to the procedure for determining the sulfate formed in the combustion (titrimetric, gravimetric, or alternate gravimetric), the data for the two samples was subdivided and compared statistically. The F test (2, 4) was used as a measure of the relative precision of the results from two methods and Student's t test (2, 4) was used to compare the accuracies by determining whether or not the difference between \bar{x} values for two methods was significant:

$$F = \frac{(s_{\bar{x}})_a^2}{(s_{\bar{x}})_b^2}$$

where $(s_{\bar{x}})_a^2$ is always the larger value.

$$t = \bar{x}_a - \bar{x}_b \sqrt{\frac{n_a n_b (n_a + n_b - 2)}{(n_a + n_b) [\Sigma(\bar{x} - \bar{x})_a^2 + \Sigma(\bar{x} - \bar{x})_b^2]}}$$

where \bar{x}_a and \bar{x}_b are the grand means for groups a and b ; n_a and n_b are the number of values in the two groups; and $\Sigma(\bar{x} - \bar{x})_{a \& b}^2$ are the sum of squares of the differences between the individual \bar{x} 's and \bar{x} for groups a and b .

These two tests were applied to the data for the Carius and catalytic methods for both samples and no significant differences in either accuracy or precision were indicated.

Comparison of the data obtained by the titrimetric and gravimetric procedures for determining sulfate, however, showed a highly significant difference in precision between the two methods, since the F values of 7.30 and 5.63 for samples 1 and 2 were even greater than 5.41, the critical F at the 2 per cent level. The lower precision of the gravimetric method shows the need for refinement of this procedure. This is essential because the titrimetric method is not applicable when the sample contains phosphorus. The difference between the means for these two sulfate methods was not critical for either sample. The alternate gravimetric results were not used in any comparisons because too few data were available. The agreement between the results obtained by those collaborators who used the alternate method, however, was good, but the \bar{x} value for sample 1 was 0.19 per cent higher than the theoretical value.

The overall $s_{\bar{x}}$ for sample 1 (0.17 per cent) was sufficiently greater than

that for sample 2 to give an F value of 2.44 which is greater than 2.09, the critical F at the 10 per cent level. Thus better precision was obtained with the more heat-stable sulfanilamide than with the more easily volatilized benzyl-isothioureahydrochloride.

The results obtained in both the 1951 (1) and 1952 studies on micro methods for determining sulfur are summarized in Table 3. Statistical comparisons showed no significant differences in accuracy or precision

TABLE 3.—Comparison of 1951 and 1952 data on sulfur determination

	BENZYL-ISOTHIUREA HYDROCHLORIDE					SULFANILAMIDE			
		n	\bar{x}	s_x	s_y	n	\bar{x}	s_x	s_y
Carius method	(1951)	7	15.79	0.12	0.12	7	18.57	0.21	0.19
	(1952)	11	15.81	0.17	0.12	11	18.59	0.13	0.10
Catalytic combustion method	(1951)	9	15.87	0.14	0.12	11	18.60	0.13	0.11
	(1952)	10	15.79	0.16	0.11	11	18.66	0.08	0.11

between the results obtained in 1951 and 1952. In 1951 the collaborators used methods then in use in their laboratory, whereas in 1952, they used the procedures described above.

CONCLUSIONS

1. The proposed Carius and catalytic combustion methods are equally satisfactory for determining sulfur.
2. The titrimetric determination of the sulfate formed by either combustion method is more precise than the gravimetric determination even though the accuracies are not significantly different.
3. The collaborators obtained as accurate and precise results by the above procedures as by the individual laboratory methods used in the previous study.

RECOMMENDATIONS*

The Associate Referee recommends—

- (1) That both the Carius and catalytic combustion procedures be adopted as first action.
- (2) That the titrimetric method for determining the sulfate formed by the two combustion procedures be adopted, first action.
- (3) That further work be done to improve the precision of the gravimetric method which is required for samples containing phosphorus.

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* For report of Subcommittee C and action of the Association, see *This Journal*, **36**, 58 (1953).

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